Liquid Extraction For Residual Solvents

1.0 Scope and Application

1.1 This method was adapted from the EPA Method 3585 Waste Dilution For Volatile Organics.

1.2 This method describes a liquid extraction of residual solvents from cannabis product samples prior to direct injection analysis. It is designed for use in conjunction with GC or GC/MS analysis of cannabis that may contain organic chemicals at a concentration greater than 1 mg/kg and that are soluble in the dilution solvent. This method has adequate sensitivity to determine the regulatory concentrations of Washington cannabis products.

1.3 This method may be used with n-hexadecane for direct injection of target volatiles in oily matrices.

1.4 Use of a 1 - 2 µL injection of a 1:1 dilution can be used to provide detection limits of 0.5 ppm for volatile target analytes with a sensitive GC/MS.

1.5 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 Summary of the Method

2.1 Highly contaminated or highly complex samples may be diluted prior to analysis for volatiles using direct injection.

2.2 One gram of sample is weighed into a capped tube or volumetric flask. The sample is diluted to 2.0 - 10.0 mL with n-hexadecane or other appropriate solvent.

2.3 Diluted samples are injected into the GC or GC/MS for analysis.

3.0 Inferences

3.1 Use of a direct injection procedure will result in considerable contamination of injection ports, injection port liners, GC columns, and detectors. A Pyrex® wool plug should be placed into the injection port liner and the liner should be changed after every 12 hours of sample analysis.

3.2 The solvent used for waste dilution may contain volatile contaminants that could interfere with analyses.

3.2.1 *n*-Hexadecane elutes after target volatiles. However, volatile impurities in n-hexadecane may interfere with analyses.

3.2.2 Each lot of n-hexadecane (or any other solvent used for dilution) must be analyzed for impurities prior to use.

3.3 The presence of methanol and other oxygenated solvents in samples may lead to baseline humps that interfere with qualitative and quantitative analysis of early eluting target analytes when direct injection is employed.

4.0 Apparatus and Materials

4.1 Glass scintillation vials: At least 20-mL, with polytetrafluoroethylene (PTFE) — or aluminum foil-lined screw-cap, or equivalent.

4.2 Spatula: Stainless steel or PTFE.

4.3 Balance: Capable of weighing 100 g to the nearest 0.01 g.

4.4 Vials and caps: 2-mL, for GC autosampler.

4.5 The Disposable pipets: Pasteur.

4.6 Test tube rack

4.7 Pyrex® glass wool.

4.8 Volumetric flasks, Class A: 2- or 10-mL (optional)

4.9 Direct injection liner (HP catalogue #18740-80200 or equivalent): Modify with a 1-cm plug of Pyrex® wool placed approximately 50-60 mm down the length of the injection port (towards the oven). A 0.53 mm ID column is mounted 1 cm into the liner from the oven side of the injection port, according to manufacturer's specifications. Figure 1 is an example of the placement of the glass wool plug in the liner.

Figure 1: Modified Injector. Measurements in mm.

5.0 Reagents

n-Hexadecane, n-C16H34 - Pesticide quality or equivalent.

6.0 Sample Collection, Preservation, and Holding

6.1 Sample collection, preservation and storage requirements may vary by program and may be specified in a regulation or project planning document that requires compliance monitoring for a given contaminant. Where such requirements are specified in the regulation, follow those requirements. In the absence of specific regulatory requirements, use the following information as guidance in determining an appropriate plan for sample collection, preservation and storage prior to sample collection and analysis.

6.2 All samples should be stored in capped vials at ≤6 °C in an area free of solvent fumes. If any evidence of leakage is found, the sample can be considered corrupted and should be discarded.

6.3 Sample storage

6.3.1 Samples shall be stored at ≤6 °C until analysis in order to limit evaporative loss of the analytes, reduce the ability of the analytes to react with the glass walls of the sampling container and further hinder sample biodegradation. Cannabis samples in VOA vials with no headspace should not be frozen, but subsamples added to prepared headspace vials may be frozen, provided the integrity of the container seal is maintained. The sample storage area should be free of organic solvent vapors.

6.3.2 All samples shall be analyzed within 14 days of collection or sooner if labile compounds are target analytes.

7.0 Procedure

7.1 Samples consisting of multiple phases must be prepared by the phase separation method before extraction. The oil phase is prepared as outlined below.

7.2 The sample dilution may be performed in a 2- or 10-mL volumetric flask. If disposable glassware is preferred, the 10-dram vial may be calibrated for use. Pipet 2.0 mL of the solvent into the vial and mark the bottom of the meniscus. Discard this solvent. Dry the vial.

7.3 Transfer approximately 1 g of the oil phase of the sample to a vial or volumetric flask (record weight to the nearest 0.1 g). Wipe the mouth of the vial with a tissue to remove any sample material. Cap the vial before proceeding with the next sample to avoid any cross-contamination.

7.4 Immediately dilute to volume with n-hexadecane or other appropriate solvent. The choice of solvents is dependent on the nature of the target analytes. n-Hexadecane is late eluting and, therefore, presents no solvent interference for the majority of volatile organics.

7.5 Add surrogate spiking solution, if required, for the analytical method to be employed.

7.6 Cap and shake the sample for 2 minutes.

7.7 The extract is ready for analysis by GC or GC/MS Methods.

8.0 Quality Control

8.1 Refer to the analytical method to be employed, for specific quality control procedures.

8.2 Each time samples are prepared and analyzed, and when there is a change in reagents, a reagent blank should be prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory contamination. Any reagent blanks, matrix spike samples, or replicate samples should be subjected to exactly the same analytical procedures as those used on actual samples.

8.3 Standard quality assurance practices should be used with this method. Field duplicates should be collected to validate the precision of the sampling technique. Each analysis batch of 20 or fewer samples must contain: a reagent blank; either a matrix spike/matrix spike duplicate or a matrix spike and duplicate sample analysis; and a laboratory control sample, unless the determinative method provides other guidance.

8.4 Surrogates should be added to all samples when specified in the appropriate determinative method.

9.0 Method Performance

Refer to the determinative methods for performance data.

10.0 References

10.1 Marsden, P.J., Colby, B.N., and Helms, C.L., “Determining TCLP Volatiles at Regulatory Levels in Waste Oil,” Proceedings of the Eighth Annual Waste Testing and Quality Assurance Symposium, July 1992.

11.0 Acknowledgements

The above method was adapted from the EPA Method 3585 Waste Dilution For Volatile Organics for the Cannabis Laboratory Analysis Standards Program to meet the recommendations of the Cannabis Science Task Force as a standardized method for determining residual solvents for certified cannabis laboratories in the state of Washington.